GUIDELINES AND CRITERIA FOR HIGH RESOLUTION MASS SPECTROMETRIC ANALYSIS

English version

For a sample to be analysed the following criteria have to be accomplished:

A mass spectrometry request form for each sample has to be handed in along with the sample prior analysis. 1: The sample and request form should be handed in personally 3045 (MS laboratory) or 2: The sample should be put in the freezer/refrigerator in room 3B8b and the form in the box next to the refrigerator. The sample will then be put in line and analysed as soon as possible according the queue. (The form can be downloaded from http://kjinfo.b.uib.no/instrumenter/ms/analysis-request-form/ or supplied by the MS laboratory). For samples, that for some reasons (chemically) can not be stored in solution (for a short period of time), specific appointments will be made. The user will in such cases be ask to hand in the sample (at the MS laboratory, 3045) at a given analysis time. The user will be notified minimum two days in advantage.

One form for each sample and each form must contain the following information about the sample:

- Name/supervisor
- E-mail address
- Room
- Date for hand in
- Sample ID (typically your initials, synthesis nr/fraction nr etc.)
- Specify the sample content (if is a pure/purified sample or if it contains more than one compound). *As a general rule the instrument should not bee used for analysis of crude samples/products*
- Specify solvent
- Specify expected exact mass (using lowest mono isotopic mass for elements containing isotopes). If molecular mass is unknown specify excepted mass range for the compound(s).

- Elemental composition for the molecule (e.g $C_xH_yBr_z$) is a minimum. If a molecular structure is needed (for elucidation or other purposes), attached this on an additional paper along with the request form.
- Specify how the sample should be handled and stored
- Specify the requested analysis (make sure you have the best suited analysis for your sample)
- Specify the requested polarity (make sure you have the best suited polarity for the ionisation of your molecule)
- Add any other information that could be valuable for the analysis of your sample

✓ The sample should be provided as a solution prepared in proper glass vials (see below). The vial should be filled more than half full.

Tip; Wash the glass vials with an organic solvent like e.g. acetone/methanol prior sample preparation in order to diminish background ions from impurities and detergents. Avoid plastic/polypropylene tubes e.g. Eppendorf tubes.

✓ To maintain high quality data acquisition it is furthest important that samples are prepared at correct concentrations (see below). For those who do no follow the rules for sample concentration, they may be held responsible for destruction of the MCP-detector.

✓ The samples must be filtered. Minimum through a pore size of 0.45 μm. (Many syringe type filters creates a lot of back ground ions visible by the MS detector. In many cases the filters needs to be washed before filtration of samples. *Recommended: Chromacol, Econofil 4-SF-45(Nylon) supplied by Teknolab. Available in the department store.*

✓ Avoid preparing samples from compounds that has previously been in contact with deuterated solvents (NMR samples)

✓ The vials must be labelled properly. Avoid label stickers or tape that can block the vials in the auto sampler. Write directly on the vial with a readable letter

DART analysis;

- Concentration: ~ 10 μg/mL. (For a compound with molecular weight 300-400 u)
- Best solvents for DART: Methanol, acetonitrile, dichloromethane, water (use p.a quality or better).
- Glass vials with screw cap suitable for auto sampler injection.
  PTFE/silicone septum (bonded to cap). Available in the department store.

Electrospray analysis (ESI) – direct infusion;

- Concentration: ~ 5-10 μg/mL. (For a compound with molecular weight 300-400 u)
- Best solvents for ESI+:Methanol, acetonitrile, water (use p.a quality or better).
  Best solvents for ESI-: Acetonitrile (use p.a quality or better). Avoid any traces of acid.
- Glass vials with screw cap suitable for auto sampler injection.
  PTFE/silicone septum (bonded to cap). Available in the department store.

Atmospheric pressure chemical ionization (APCI) – direct infusion;

- Concentration: ~ 5-10 μg/mL. (For a compound with molecular weight 300-400 u)
- Best solvents for APCI: Hexane, Acetonitrile, dichloromethane (use p.a quality or better).
- Glass vials with screw cap suitable for auto sampler injection.
  PTFE/silicone septum (bonded to cap). Available in the department store.

GC-MS (EI/CI);

- Concentration: ~5 μg/mL of main component/s. (For a compound with molecular weight 300-400 u)
- Avoid low boiling solvents if possible. Suitable solvents are; Dichloromethane, methanol (avoid Sigma 34860N Chromasolv if possible), hexanes, ethyl acetate (use p.a quality or better).
- Glass vials with screw cap suitable for auto sampler injection.
  PTFE/silicone septum (bonded to cap). Available in the department store.

LC-MS (ESI);

- Concentration: ~ 10 µg/mL of main component/s. (For a compound with molecular weight 300-400 u)
- Best solvents; use mobile phase if possible, if not mixtures of acetonitrile, methanol, water (use p.a quality or better).
- Glass vials with screw cap suitable for auto sampler injection.
  PTFE/silicone septum (bonded to cap). Available in the department store.

LC-MS (APCI);

- Concentration: ~ 10 µg/mL of main component/s. (For a compound with molecular weight 300-400 u)
- Best solvents; use mobile phase if possible, if not, use medium to non polar organic solvents (use p.a quality or better).
- Glass vials with screw cap suitable for auto sampler injection.
  PTFE/silicone septum (bonded to cap). Available in the department store.

Additional for LC-HRMS and GC-HRMS samples;

✓ Along with the analysis request copies of original LC/GC chromatograms should be given including all method details (column, oven temperature program, mobile phase, gradients, injection volume etc etc).

✓ Mobile phases should also be supplied by the user

For additional questions regarding mass spectrometric analysis contact;
Bjarte.Holmelid@kj.uib.no, (room 3B3b or 3045 (MS-laboratory))